

Appendix J. Chemical Analytical Method
1,3-dichloropropene - sorbent tubes
California Department of Food and Agriculture Laboratory

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Determination of Telone in Air Samples Using Charcoal Tubes

Scope: This method was developed to analyze Telone (1, 3-dichloropropene) absorbed on SKC 226-09 charcoal air sample tubes. The reporting limit is 0.1 µg/sample.

Principle: Telone, $C_3H_4Cl_2$, absorbed from the air onto activated charcoal is extracted from the charcoal with 10% carbon disulfide in hexane. Telone is subsequently quantitated using a gas chromatograph equipped with a DB-624 capillary column and an electron capture detector⁽¹⁾ (ECD).

Reagents and equipment:

Reagents:

1. Carbon Disulfide (CS_2), OmniSolv[®], HPLC grade
2. Hexane, Fisher, pesticide grade
3. Telone (CAS registry # 542-75-6); Standards in acetone: obtained from Standards Repository, CAC, 3292 Meadowview Rd, Sacramento, CA, 95832
4. Charcoal tubes - 200/400 mg, SKC[®] #226-09, Lot 120
SKC, 334 Valley View Road, Eighty Four, PA, 15330, phone: (800) 752-9378

Equipment:

1. Amber serum bottles, 10 mL, with aluminum seals (tear off)
2. Amber patch autosampler vials, with screw cap and teflon/silicone septa
3. Assorted pipettes
4. Small triangular file
5. G-10 Gyrotory[®] Shaker, New Brunswick Scientific Co., Inc, N.J. USA
6. Forceps
7. Micro-syringes
8. HP 5890 GC with ECD

Analysis:

Sample Extraction:

1. Remove samples from refrigerated storage (freezer). Allow samples to stand at ambient temperature for 15-30 minutes.
2. Place a sheet of white paper under charcoal tube to catch spills.

Sample Extraction: (continued)

3. Remove caps from sample tube. Score the glass tube with a file in front of the glass wool plug and break the tube.
4. Pipette 3.0 mL of carbon disulfide/hexane (10:90 v/v) solvent mixture into a labeled amber glass bottle.
5. Using forceps, remove the spring wire and place it in the amber glass bottle.
6. Placing the large open end of the sample tube into the mouth of the amber bottle, insert a Pasteur pipette from the opposite end and push the whole contents of the tube into the amber bottle. Immediately cap the bottle.
CAUTION: HEAT WILL BE GENERATED AND LOSS OF TELONE MAY OCCUR.
7. Place bottle on G-10 Gyrotory shaker and shake for 10 minutes at 200 rpm.
8. Transfer sample extract into an amber autosampler vial. Store vial at 4 °C until analysis.
9. Determine Telone using gas chromatographic (GC) method below.
10. If total peak height for Telone (cis + trans) is greater than that of the highest standard, dilute sample, mix thoroughly and reinject.

Preparation of the Blank:

1. Score SKC 226-09 tube with a file in front of the glass wool plug and break the tube.
2. Score tip of the opposite end of the same tube and break the end.
3. Follow steps five to ten under *Sample Extraction*.

Preparation of the Spike:

1. Score SKC 226-09 tube with a file in front of the glass wool plug and break the tube.
2. Score tip of the opposite end of the same tube and break the end.
3. Draw into a micro-syringe 10 µL of 10% CS₂ in hexane Telone spiking solution. Place syringe needle past the glass wool plug and about 1 cm into the charcoal. Add spiking solution onto the charcoal.
4. Wait 1 to 3 minutes.
5. Follow steps five to ten under *Sample Extraction*.

Instrument Conditions:

GC:	Hewlett Packard 5890 Series II equipped with ECD
Column:	J&W Scientific DB-624, 30 m, 0.32 mm i.d., 1.8 µm film thickness
Gas flow rate:	Helium: 1.7 mL/minutes, 8 psi Splitter: 37 mL/minutes, open at 0.8 minutes
Injector:	250 °C
Detector:	350 °C
Oven temperature:	Initial temperature: 40 °C held for 1 min Rate 1: 50 °C/min to 70 °C held for 1min Rate 2: 1 °C/min to 82 °C held for 0 min Final temperature: 50 °C/min to 225 °C held for 5 min
Total time	24.46 minutes
Injection volume:	2 µL
Retention Time:	cis-1,3-dichloropropene=10.5±0.1 minutes trans-1,3-dichloropropene=12.3±0.1 minutes

Calculations:

Calculate the total amount of Telone in a charcoal tube using a linear regression equation. Use five to ten levels of standard to create the calibration curve. Calculate cis and trans separately and add them together for total Telone.

$$\mu\text{g/sample} = (\mu\text{g Telone}_{\text{cis+trans}}/\text{mL}) (\text{sample volume, mL}) (\text{dilution factor})$$

Method Performance:*Quality Control:*

1. A five to ten point standard curve (0.015, 0.025, 0.05, 0.1, 0.25, 0.5, 1.0, 2.5, 5.0, and 10.0 ng/ μL), was obtained at the beginning and the end of each set of samples to create a cis and trans calibration curve. These calibration curves were used to calculate results and check instrument performance.
2. A sample set is comprised of 10 samples or less, a blank and two spike. Standards and samples were injected twice sequentially to insure reliability for the analysis.

Recovery Data:

Validation: Method validation was done by spiking charcoal tubes with ten microliters of three different levels of standard (25, 251.4 and 8838.1 ng/ μL). Five separate sets were analyzed. A matrix blank was also analyzed which each set. The recovery data is summarized in the table below:

Recoveries of Telone

Spike level (μg)	set 1 (%)	set 2 (%)	set 3 (%)	set 4 (%)	set 5 (%)	Avg. (%)	Standard Deviation (based on % recovery)
0.25	96	88	92	84	84	89	5.2
2.5	96	96	106	103	95	99	5.0
8	91	88	95	97	90	92	3.7

Method Detection Limit (MDL):

Method detection limit refers to the lowest concentration of analyte that a method can detect reliably in either a sample or blank. To determine the MDL, seven charcoal tubes were spiked separately with 0.15 μg of Telone by following the procedure in the sample extraction section. These spiked samples along with a blank were then analyzed using the described method. The standard deviation derived from the seven spiked samples was used to calculate the MDL using the following equation⁽²⁾:

$$\text{MDL} = t S$$

where

t: is the Student 't' value for the 99% confidence level with n-1 degree of freedom (in this case t is 3.143)

S: is the standard deviation obtained from the recovery (μg) of replicate analyses.

Method Detection Limit: (continued)

Sample No.	µg Spiked	µg Recovered
1	0.15	0.107
2	0.15	0.108
3	0.15	0.102
4	0.15	0.106
5	0.15	0.124
6	0.15	0.127
7	0.15	0.106

Average µg/sample recovered = 0.111

Standard Deviation = 0.010

The calculated MDL for Telone is 0.031 µg/sample.

Reporting Limit:

Reporting limit refers to the level above which quantitative results may be obtained. In this method, the reporting limit was set at 0.10 µg/sample.

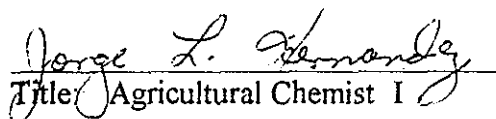
Discussion:

The Air Resources Board Standard Operating Procedure for the Analysis of Telone in Ambient Air⁽¹⁾ has been followed as closely as possible to develop this method. Additional input came from Mario Fracchia at Department of Health Services.

REFERENCE:

1. State of California Air Resources Board Monitoring and Laboratory Division/EED, *Standard Operating Procedure for the Analysis of Telone (1,3-dichloropropene) in Ambient Air*
2. EPA, Appendix B to Part 136, 40 CFR Ch.1, pp. 569-571, 7-1-93 Edition
3. Robert B. Lew, *Method EM 59.5, Determination of Telone (1,3-dichloropropene) Absorbed on Charcoal*, Center for Analytical Chemistry, Environmental Monitoring Section, January 1995

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